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Characterization of Chemical and Physical Properties of Distillers Dried Grain with Solubles (DDGS) for Value Added Uses

Rumela Bhadra
Graduate Research Assistant, Department of Agricultural and Biosystems Engineering, South Dakota State University, Brookings, SD 57007

K. Muthukumarappan, Ph.D.
Professor, Department of Agricultural and Biosystems Engineering, South Dakota State University, Brookings, SD 57007

Kurt. A. Rosentrater, Ph.D.
Bioprocess Engineer, USDA ARS North Central Agricultural Research Laboratory, 2923 Medary Avenue Brookings, SD 57007

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Abstract. One of the fastest growing industries in the United States is the fuel ethanol industry. Since 2000 there has been an increase of more than 300%. There was production of 4.9 billion gallons of ethanol in 2006. The major coproducts from this industry include Distillers Dried Grains with Solubles (DDGS) and carbon dioxide. DDGS is used as a livestock feed since it contains high quantities of protein, fiber, amino acids, and other nutrients. The goal of this study was to quantify various chemical and physical properties of DDGS, Distillers Wet Grains (DWG), and Distillers Dried Grain (DDG) from several plants in South Dakota during fall and winter 2006-2007. Chemical properties included crude ash, Neutral Detergent Fiber (NDF), Acid Detergent Fiber (ADF), crude fiber (CF), crude protein, crude fat and total starch content in each of the samples. Physical properties included moisture content, water activity, bulk density, thermal properties, color (L*, a*, b*) and angle of repose. We also conducted image analysis and particle size determination of the DDGS. The carbon groups in the DDGS samples were determined using NMR spectroscopy. Results from this study showed several possibilities for using DDGS as alternatives other than animal feed. Possibilities include degrading it with suitable enzymes and producing additional ethanol, producing value added compounds, using it as human food additives, or even using as inert fillers for biocomposites.

Keywords. Chemical properties, coproducts, DDG, DDGS, DWG, ethanol, fiber, NMR spectroscopy, physical properties.

Introduction

The potential increase in the demand of ethanol as a fuel additive and as an alternate fuel has caused a radical transformation in the growth of the corn distillers industry throughout the USA. According to a recent Renewable Fuel Association report, about 10.74 billion bushels of corn was produced in 2006, out of which 1.8 billion corn bushels went to the ethanol industry for bioethanol production (RFA, 2007). Thus, the fuel ethanol industry represented 17% of the total US corn production. In 2006, 110 manufacturing plants in the US had a total output production capacity of nearly 4.9 billion gal of ethanol (RFA, 2007). No fewer than 15 new biorefineries will be coming online within a year. It was estimated that between 13 and 12 million metric tons of DDGS was produced in 2006. The amount of corn used for the ethanol fermentation process has increased 17 fold during past 20 years, as has the quantity of the coproducts.

The processing of ethanol from corn is mainly classified into two types. One is wet milling, where ethanol is obtained in large volumes. These facilities are generally corporate owned and require more capital. In the wet milling process, starch is isolated in pure form due to fractionation of the corn into starch, fiber, germ and protein. Wet milling requires sophistication and yields coproducts such as Corn Gluten Feed (CGF), germ meal, Corn Gluten Meal (CGM), and crude corn oil. The other process is dry milling. According to the RFA 2006 report, 82% production of ethanol comes from dry milling, while only 18% comes from wet milling. The dry milling process has no fractionation and the primary coproduct is known as distillers dried grains with solubles (DDGS). It is the left over, or residual, non-fermentable components after corn fermentation in ethanol processing. The residuals are often pressed to remove the excess water via centrifugation; once the removal of water is completed they are mixed with soluble materials and then dried. This final product is termed distillers dried grains with solubles, DDGS (Rosentrater, 2005). The solubles are often referred to as “syrup” in the industry. They are high in vitamins, fat, and protein, but low in fiber content. Syrup yields a digestible energy value approximately 91% of that of raw corn (Buchheit, 2002; Cruz et al., 2005). It typically contains approximately 28% to 46% dry matter, 6% to 21% (db) fat, 18% to 22% (db) protein, and 9% to 12% (db) minerals (Belyea et al., 1998; Schingoethe, 2001). The dry milling production process usually consists of several steps: grinding, cooking, liquefying, saccharifying, fermenting, and distilling the corn grain (Rosentrater, 2005). More details about this process can be obtained from Tibelius (1996), Weigel et al. (1997), Dein et al. (2003), and Jaques et al. (2003).

DDGS is used exclusively as livestock feed. The composition of DDGS, nutritive value, shelf life, and transportability parameters are therefore vital in terms of feed quality. Based on these parameters, and the values of their properties, the sale of DDGS, economic viability of each ethanol plant, and other operational factors change accordingly. Thus, research has been done related to the nutritional properties (Spiehs et al., 2002), physical properties (Rosentrater, 2005), and flowability of DDGS (Ganesan et al., 2006).

Understanding these facts, various studies have been conducted on DDGS, yet there are still more areas in which DDGS could be used as value added products in addition to feed. Removal of fiber from DDGS (Singh et al., 2001b), biodiesel production from corn oil, biomass gasification, and cellulosic degradation of DDGS for further ethanol production, to name a few. However to address these new areas, a complete understanding of DDGS is lacking. Thus, the objective of this study was to quantify various physical and chemical properties of DDGS in order to establish a baseline from which to pursue these options.

Materials and Method

Sample Collection

In this study, samples of DDGS and DWG were collected from three commercial ethanol plants (denoted here as Plant I, Plant II, and Plant III) in the state of South Dakota. Sampling was done in two batches (denoted as Batch I and Batch II), consisting of two different time periods. One batch of DDGS and DWG were collected in the month of September, 2006 and the other in the month of December, 2006. The DDGS samples were stored at room temperature (24°C ± 1°C), while the DWG samples were stored under refrigerated condition (5°C ± 1°C). A third sample, DDG, was collected from only one plant and was analyzed for the same properties as the DDGS and DWG, for comparison purposes.

The samples were subjected to various physical and chemical properties. For each of the properties, for a corresponding batch and plant, five replications were taken, thus, n=30 for each property for each product stream across all plants. For determination of crude fat (% db), crude protein (% db), and total starch (% db), only two replication were taken from each plant and their corresponding batches, thus n=12. Each of the properties was studied using a completely random design. For each of the properties minimum, maximum, mean, and standard deviation was determined using Microsoft Excel, 2003 (Microsoft Corp., Redmond, WA.). A Least Significant Difference (LSD) test was performed for all the physical and chemical properties, at 95% confidence level using α=0.05, for each sample through SAS 9.1 (SAS Institute, Carry, NC) software.

Physical Properties

Moisture content was determined following ASAE standard method S352.1 (2004), using a forced convection laboratory oven (Thelco Precision, Jovan Inc., Wincester, Va.) at 103°C for 72 hours. Water activity was measured using a calibrated water activity meter (AW Sprint TH 500, Novasina, Talstrasse, Switzerland). Thermal properties (conductivity, diffusivity, and resistivity) were determined with a thermal properties meter (KD2, Decagon Devices, Pullman, WA.) that utilized the line heat source probe technique (Baghe-Khandan et al., 1981). Bulk density was measured using a standard bushel tester by the method described by USDA (1999) (Seedburo equipment Co., Chicago, IL.). Color was measured using a spectrocolorimeter (LabScan XE, Hunter Associates Laboratory, Reston, VA.) using the L-a-b opposable color scales (Hunter Associates Laboratory, 2002). Angle of repose for DDGS and DDG were determined by the method described by Mohsenin (1980), where the DDGS and DDG were allowed to fall onto a 44 mm diameter circular plate. Particle size distribution was measured using a Rotap Sieve Analyzer (model RX-29) for DDGS and DDG, and the geometric mean diameter and geometric standard deviation for each observation was calculated using ASAE standard method S319.3 (ASAE, 2004). For DDGS only, microscopic analysis was done for the particles from each sieving screen, using an Olympus SZH10 stereo microscope with a DP digital camera, followed by image analysis of the particles by Image Pro Plus software, to determine the maximum diameter, minimum diameter, area, and roundness.

Chemical Properties

Ash content was determined using method 08 – 01 (AACC, 2000). Acid Detergent Fiber (ADF), Neutral Detergent Fiber (NDF), and crude fiber analysis was done with an ANKOM fiber analyzer (Macedon, NY). Protein content was determined using method 990.03, fat content with method 920.39, (AOAC, 2003), and total starch was measured following Xiong et al. (1990). These were determined using two replications only. Nuclear magnetic resonance (NMR) spectroscopy was conducted at the Department of Chemistry and Biochemistry at South Dakota State University.

NMR spectra were only done for DDGS (all batches) and DDG (only from Batch I), using single replications.

Results and Discussion

There were significant differences observed between the values of different properties among the plants, as well as between the two batches (i.e, time periods of collection), within a single plant. The results of the statistical analysis with their mean values are given below, from Table 1 to Table 8. Table 1 and Table 5 show the significant differences among the plants for DDGS and DWG, respectively, for all the physical and chemical properties. Tables 2, 3, and 4 give the significant differences between the two batches of DDGS for various parameters, while tables 6, 7, and 8 give the significant differences between the two batches of DWG for the same parameters. Differences in the physical and chemical properties of ethanol coproducts lead to inconsistencies of the products in the market place (Ganesan et al., 2006).

Dried Distillers Grain with Solubles (DDGS)

Physical Properties

The physical properties results are shown in Table 9. The moisture content was from 3.54% db (minimum) to 8.21%, db (maximum), with a mean value of 5.07% db. This low moisture content was seen because DDGS was dried well before it was sold in the market. This value differed from moisture content data obtained by Rosentrater (2005). The moisture content was nearly half to one third in our results, compared to the findings by Rosentrater (2005). Standard deviation was found to be a little less (~1.21%) as well. Typically moisture contents less than 12% are recommended for storage, handling and transportation. Thus the moisture content was found below the marginal value, and should help in mobility and long term usage for any value added applications. Moisture content and soluble levels will affect the flowability of DDGS (Ganesan et al., 2006).

The water activity was in the range of 0.42 to 0.53, with a very low standard deviation of 0.04. The average water activity was found to be 0.48. This value differed slightly from the results of Rosentrater (2005). It represents the amount of free water available for microbial activity. The lower the value of water activity, the less prone it will be to microbial spoilage. Very low levels of water activity prevent microbial spoilage and increase the shelf life. Materials have reduced chances of bacterial, fungal, and yeast growth below water activity value of 0.7 (Barbosa-Canovas and Vega-Mercado, 1996).

Bulk density was found to range from 467.7 to 509.38 kg/m^3, with a mean value of 488.97 kg/m^3. It had a standard deviation of 14.96 kg/m^3, but a lower coefficient of variability (3.06%) The standard deviation value was less compared to that found by Rosentrater (2005).

Thermal conductivity was found to be from 0.05 to 0.07 W/m°C, with small variations (standard deviation of 0.005 W/m°C). Similarly, for thermal resistivity it was from 13.1 to 16.4 m°C/W, and thermal diffusivity was from 0.1 to 0.17 mm^2/s. The standard deviations were found to be low: 0.037 and 0.008 for resistivity and diffusivity, respectively. These values were very close to those found by Rosentrater (2005). The standard deviations were also similar to those found by Rosentrater (2005). These results were anticipated because thermal properties are inherent to the material, and thus they are less prone to variations among the plants or over time periods.

For color parameters, L was found to be in the range of 36.56 to 50.17 (mean value of 42.3), a was found to be in the range of 5.2 to 10.79 (mean value of 9.65), b was found to be in the range of 12.53 to 23.36, with an average of 20.62. There were differences in the ranges obtained in all three color scales compared to those found by Rosentrater (2005). This current study showed broader ranges, indicating much variation among the batches as well as between the processing plants. Color values may be related to the nutritional characteristics of the samples (Goihl, 1993; Ergul et al., 2003). Rosentrater (2005) has found correlations between color parameters and other physical properties: color parameters a and b were found to have high correlations with water activity and moderate correlations with thermal properties. This indicates that if DDGS is used in further processing, then nutritional characteristics and color may play a vital role.

The angle of repose was found to range from 25.7° to 47.04°, with an average value of 45.14°. The mean value was very close to that found by Rosentrater (2005), but the range was broader in our current findings. This indicates higher variability among the plants and the batches. Finer particles were obtained in our findings, which could affect the flowability of DDGS (Ganesan et al., 2005), and may affect the possibility of caking. Angle of repose gives an idea of grain structure: the higher the angle of repose value, the lower the flow rate. The difference between angle of repose values depends on the machine parameters and processing techniques used by an individual plant, which may sufficiently differ from one plant to another.

Physical properties usually provide an idea of the potential flow properties of DDGS. Particle shapes, size, edges, moisture, angle of repose, and bulk density are some of the key parameters that influence flow and transportation problems. Caking and stickiness are some of the common problems of DDGS (Ganesan et al., 2006). This caking is an added burden for the DDGS market where additional cost is necessary for breaking the consolidated particles. Chemical properties also play a vital role in the flowability.

Chemical Properties

The results for chemical properties are shown in table 12. The greatest constituent was Neutral Detergent Fiber (with overall mean value of 36.74% db), then was crude protein (overall mean value 29.93% db) followed by Acid Detergent Fiber (overall mean value of 16.2% db), crude ash (overall mean value of 12.82% db), total starch (overall mean value 11.07% db), total fat (overall mean value of 10.5% db) and crude fiber (overall mean value of 10.22% db).

Crude ash was found to range from 5.00% (db) to 21.93% (db) with mean a value of 12.82 % (db). The average ash content was higher compared to the results obtained by Spiehs et al. (2002), and our results showed a broader range of ash content values. This indicates variations in the amount of minerals among the processing plants. Since the plants were located at different places, variation among the corn types, which in turn depends on the soil properties and mineral availability, can be a possible reason for the broader range of ash content. It is also reported that the ratios of grain used and solubles added to during DDGS processing, fermentation processes, and corn types will influences the nutritional properties more than soil behavior and fertility (Spiehs et al., 2002).

Neutral Detergent Fiber (NDF) was found to range from 26.32% db to 43.50% db (mean value of 36.74% db). Acid Detergent Fiber (ADF) was found to range from 10.82% db to 20.05% db (mean value of 16.2% db). The crude fiber (CF) was found to be from 8.14% db to 12.82% db (mean value of 10.22% db). The NDF and crude fiber content are slightly higher than those found by Spiehs et al. (2002). ADF values were very similar to that found by Spiehs et al. (2002). NDF is the sum of the ADF and the hemicellulose content. ADF is composed of cellulose and lignin. This category of fibers (Neutral Detergent Fiber, Acid Detergent Fiber, and crude fiber) is generally called insoluble fiber content. Crude fiber is generally achieved by subtracting ADF value from NDF content. This subtraction is not completely accurate, but a close value to CF is reached. From the comparison study of Speihs et al. (2002), it is evident that our samples showed higher amount of hemicellulose, because they showed higher NDF and CF ratios, but similar ADF contents. Our results also indicate higher average NDF (36.74% db) value than the protein content (29.93% db), which means our DDGS samples were higher in overall cellulose, lignin, and hemicellulose content. The difference of NDF value (36.74%, db) from the ADF (10.85%) indicates the presence of higher amount of hemicellulose content in the DDGS.

Thus, these results indicate that DDGS could possibly be used with enzymatic hydrolysis to break down the insoluble fiber to yield further ethanol. The presence of cellulose, hemicellulose, and lignin could be utilized by appropriate enzymes to break down into glucose, which can be further converted by fermentation into ethanol, or can be used as glucose solely in food industries while making confectioneries, bakery products, candies, and many more.

Another interesting fact that was observed was the presence of starch in the DDGS. It is a fact that 100% of the starch is not being converted to ethanol in typical ethanol plant, but only approximately 70% of it can be, thus the rest remains in the coproduct stream. If DDGS is subjected to enzymatic degradation with cellulase and hemicellulases to yield ethanol, then the addition of amylase would also convert the left over starch; to glucose, then to ethanol. Large protein molecules and the presence of unknown inhibitory proteases can possibly inhibit the fermentation process. De-proteinization studies could reveal further aspects of this problem, and could help us in eradicating the issue of the partial starch conversion to ethanol from corn.

Thus, from the chemical properties, it appears that we should be able to use DDGS for further enzymatic degradation to yield glucose and ethanol before selling it for livestock feed. This would, in turn, bring up the overall ethanol production capacity per bushel of corn, and bring down the price of corn per bushel, thus releasing the pressure of corn production in United States while maintaining the ethanol demand. Additionally it would reduce the quantity of DDGS produced, and increase the protein content of this new coproduct stream.

NMR Spectroscopy

Nuclear magnetic resonance (NMR) provides us with the nature of the compounds present. The results of carbon partitioning through NMR spectroscopy is given in Table 16. The highest C – group was O-alkyl with an average 52.81%, then followed by alkyl (overall mean of 28.86%), aromatic (overall mean of 10.75%), carboxyl (overall mean of 7.56%), and carbonyl (overall mean of 0.11%) over all the samples, irrespective of plant and sampling time. Only one replication for each sample was done. There were differences observed in the carbon partitioning values among the plants and the time periods. For Plant III there was less variation in the carbon groups; for plant I and plant II, there were differences in the O-alkyl and alkyl values between these two plants, as well as between the two batches (time periods) within the same plant. No previous studies with NMR spectroscopy have yet been done on DDGS. These results indicate the nature of the compounds present, and the possible value added uses that could be developed from DDGS in the future.

Alkyl (CH_3O :) groups are made due to the de-protonation from the alcohol molecules (Fletcher, 1974). A higher number of alkyl groups indicates the presence of straight chain compounds, such as simple carbohydrates with possible alkoxyl groups. Aromatic compounds were not found in high amounts. Aromatic compounds are made up benzene rings, usually found in secondary plant metabolites like carotenoids, shikimic acids, plant steroids, etc. (Trevor, 1975). Thus, a relatively less percentage of these compounds would indicate that it would probably not be appropriate to use DDGS for harvesting pharmaceutical compounds such as antioxidants, carotenoids or such other value added molecules which could possibly be found in aromatic ring structures. Phytosterols, an aromatic nutraceutical compound, was previously thought to be found more in the fiber part (after fiber removal) than the original DDGS samples, because phytosterols are usually found in the cell walls and fibrous tissue. Most of these phytosterols were found to be associated with the pericarp layer (Singh et al., 2001a). But additional studies showed there was less amount of phytosterols recovered in the aspirated (fiber rich) part (Singh et al., 2001b). This indicates that such types of use may be difficult with DDGS, due to sufficient hindrance of aromatic compounds' recovery by the presence of cross linked fibers.

Biodiesel is the ester which can be made using vegetable oils, animal fats, algae, or even recycled greases. It is usually used as fuel additives in trucks and other vehicles (USDOE, 2006).

The presence of charged alkoxly groups would suggest the probability of finding such esterified compounds with the fatty acids present in DDGS (Hassner, 2002).

Presence of hydrocarbons would favor the process of gasification, conversion of DDGS biomass to gas mixture of hydrogen, methane, and carbon monoxide (USDOE, 2006). It would be more favorable to use DDGS for further ethanol conversion by enzymatic hydrolysis due the higher amounts of carbohydrate compounds founds. On the other hand, having higher O-alkyl groups, charged electrons on the oxygen molecules, could enhance the binding properties of DDGS, and thus, it should be able to be used as biocomposites or biofillers in value added products.

Particle Size and Image Analysis

The results of the image analysis are found in Table 15. There were many differences in the measured parameters (i.e., maximum diameter, minimum diameter, average area, and the roundness values) of the DDGS size fractions. For each plant, the particles were sampled from different screens (from screen no. 8 (2.38 mm) to screen no. 100 (149 μm)). Geometric mean diameter, or median size (d_{gw}), was highest in Plant III, then Plant I, and Plant II. Standard geometric deviation (S_{log}) was greater in Plant I, followed by Plant III, and then Plant II. Particle diameters ranged from 6.87 mm to 0.17 mm for Plant I and II, while for Plant III it was found to range from 29.00 mm to 0.36 mm.The greatest area was found in particles from Plant III (16.70 mm^2) and the for the other two plants, the value of their greatest areas were quite similar to each other (~5.50 mm^2).

Roundness is defined as the degree of abrasion of a grain particle as shown by the sharpness of its edges and the corners. By 'roundness', we mean either the sphericity of a three dimensional body, or the circularity of a two dimensional figure (Cox, 1927). The sphericity of a three dimensional body may be expressed by the degree to which the ratio of its volume to its surface approaches the same ratio as a sphere. In grain particles it is measured by the degree to which the ratio of the area to the circumference approaches the same ratio for a circle. Expressed mathematically:

$$K= A / (P)^2 \tag{1}$$

This is ¼ п for a circle. Therefore multiplying equation 1 by 4 п, we have:

$$K= A *4\pi/ (Pr)^2 \tag{2}$$

Where A is the area, P is the perimeter, and K is the constant that depends on the shape on the particle (K =1 for a circle or sphere, but less than 1 for any other shape). K represents the percentage ratio (%) that the area of the figure holds to the area of a circle with same perimeter (Cox, 1927). For example, if K is 0.78 of a square, it means that a square contains just 78% of the area that a circle with same perimeter would contain. Higher the roundness value, the more regular the edges of the object.

The highest roundness was found in Plant I (64.30%), and then in Plant III (56.61%), followed by Plant II (31.30%). From these findings, we can say that the DDGS from Plant I and Plant III have more round edges than Plant II. A roundness ratio from 96% to 80% is called a "well rounded object", 95% to 74% is called a "fairly well rounded object", and 83% to 60% roundness value objects are known as "angular" (Cox, 1927).

More irregularity on the edges, with sufficient roughness, would possibly favor using particles with binders. The particles from the Plant II have lower roundness ratios and thus have sufficient roughness on the edges. Large size particles were obtained from Plant III with the highest area (16.70 mm^2) and maximum diameter (29.00 mm); large particle sizes favor using DDGS as biocomposites. Fairly large size particles, but less roundness values (56.61%), would not favor the essential lock and key mechanisms required to form composites.

Large DDGS particles could lower the efficiency of further chemical processing, however, if such technology is going to be used down the line. Particles with smaller area and dimensions were also obtained. Those particles had higher surface to volume ratios, which may favor further bioprocessing, but not for using it as biocomposite materials. Of course, particle sizes can be reduced using grinding and milling.

Thus, we need to examine and pursue promising avenues to utilize DDGS for value added uses. Image analysis and particle size determination can address these issues only partially. Differences were observed between the plants, time periods within each plant were not examined. This seems to be a logical next step, because the grain size and particle shape depend very much on the processing machines used by the plants, and these differ from one plant to other plant, and will vary over time

Distillers Dried Grain (DDG)

Physical Properties

The results of the physical properties of DDG are given in Table 10. Moisture content of DDG was found to range from 2.17% db to 2.71% db. This was much less than the DDGS' moisture content, which was found to range from 3.54% to 8.21%, and was less than the level of moisture content for safe storage, which is ~ 12%. With increases in moisture content, there have been problems noted in the flowability of DDGS (Ganesan et al., 2005). Our results indicate that DDG should be suitable for storage, flowability, and handling.

Water activity was found to be constant at 0.42, with no standard deviations. This value was less than that of DDGS water activity (0.48). Low water activity will prevent the microbial spoilage and should ease storage and handling of DDG (Rosentrater, 2005).

The bulk density was found to range from 467.30 to 482.24 kg/m^3. The bulk density of DDG was found to be less than that of DDGS (ranges from 467.7 to 509.83 kg/m^3). DDGS does have solubles, but DDG does not, which could explain those differences. However, it has been found that DDG can have up to initially 8% solubles (Ganesan et al., 2005).

Thermal conductivity was found to range from 0.06 to 0.08 W/m°C, resistivity ranged from 12.8 to 17.9 m°C/W, and diffusivity was found to be within 0.14 to 0.17 mm^2/s. The ranges of the thermal properties for DDG were very near those obtained for DDGS. Variation observed was much less for all the thermal properties. Thus, like DDGS, DDG should be suitable for subsequent chemical/biochemical process.

L value was found to range from 52.43 to 54.82, a was from 5.9 to 6.72, and b was from 23.76 to 24.29. The values of the color scales differed substantially from those obtained from DDGS. This may be an indication that DDG has different nutritional characteristics than DDGS, due to the absence of substantial levels of solubles in DDG (Goihl, 1993; Ergul et al., 2003).

The angle of repose was found to range from 20.32° to 29.9°. The maximum margin was found to be lower in DDG than DDGS. Therefore DDG was potentially more free-flowing than DDGS. This is possible because DDG does not have the soluble fat layers on its surface.

Chemical Properties

The results for the chemical properties of DDG are shown in table 13. The greatest constituent was NDF (overall average value of 31.43% db), then was crude protein (overall average value of 30.9% db), followed by ADF (overall average value of 28.69% db), crude fiber (overall average value of 12.33% db), total starch (overall average value of 11.01% db), crude ash (overall average value of 10.91% db), and crude fat (overall average value of 8.9% db).

The NDF content was found to range from 29.39% db to 33.82% db. Thus, the NDF contents were very close to those found in DDGS. Average NDF value of DDG (31.43% db) was a little less than the NDF content of DDGS (36.74% db). The ADF value was found to range from 27.99% db to 29.42% db. This range of ADF content in DDG was almost double the ADF content in DDGS. Since we know NDF is the sum of ADF and hemicellulose, the higher ADF value indicates less amounts of hemicellulose compared to DDGS, and higher cellulose and lignin values. Thus, degradation with only hemicellulase enzymes would not yield sufficient glucose molecules if DDG is subjected to a single enzymatic degradation.

Crude protein of DDG was found to be in the range of 30.6% db to 31.2% db. It was slightly higher than found in DDGS. Since the DDGS is formed by incorporation of solubles, which are essentially fat compounds, the protein compositions did not vary much between DDGS and DDG.

The total starch content range was found to be from 9.91% db to 12.83% db. This range was quite close to DDGS, but the maximum margin was higher in DDGS than DDG. Again, due to similar processing techniques, DDG and DDGS had similar starch levels.

The fat content of DDG was found to be in the range of 8.1% db to 9.7% db. As suspected, the fat content was lower in DDG than DDGS because there were no solubles added.

NMR Spectroscopy

The carbon partitioning of DDG, shown in table 17, was quite similar to the DDGS. The highest ratio was found in the O-alkyl group (50.08%), followed by alkyl (27.46%), aromatic (15.15%), carboxyl (7.11%), and carbonyl (0.18%). Thus, like DDGS, DDG could also be it used for biocomposites and or enzymatic hydrolysis, due to high carbohydrates and charged alky groups. The aromatic group ratio was found to be a bit higher in DDG than DDGS. Follow up studies should examine DDG from multiple plants over time.

Distillers Wet Grain (DWG)

Physical Properties

The values of the physical properties of DWG are given in table 11. The moisture content of DWG ranged from 22.85% db to 43.66% db, and was very much higher than DDGS, which was quite expected because DWG is the wet part of Distillers Dried Grain (DDG). There were differences in the moisture content among the plants as well between the batches. Higher moisture content in DWG creates difficulties in transportation, storage, and shipping over long distances. The water activity was found to be around 0.96 (range from 0.93 to 0.99). Higher water activity will facilitate rapid microbial spoilage to the samples. Water activity around 0.9 would foster mold and other fungal growth. DWG is then not at all found to be favorable for long term storage or transportation.

Bulk density of DWG was found to range from 794.55 to 1107.6 kg/m^3. The bulk density was found to be more in DWG than DDGS. Higher bulk density was due to inclusion of water molecules in the DWG. This will make DWG difficult to handle and work with in future technologies. DWG was found to have greater thermal conductivity (average value of 0.12 W/m°C) and thermal diffusivity (average value of 0.11 mm^2/s) than DDGS. This was also due to presence of high water levels, which can increase thermal conductivity. Thus, unlike DDGS, DWG may not be readily effective in further chemical processing or other value added uses as it shows higher conductivity, water activity, and bulk density. Overall average L, a, b values were found to be 50.94, 6.91, and 8.88, respectively.

Chemical Properties

The chemical property results are provided in table 14. There was the presence of fairly high amount of fiber content in DWG. The greatest constituent was NDF (overall average value of 33.80% db), then followed by crude protein (overall average value of 28.62% db), ADF (overall average value of 14.22% db), crude ash (overall average value of 13.31% db), crude fiber (overall average value of 12.04% db), total starch (overall average value of 11.24% db), and crude fat (overall average value of 11.12% db). DWG showed almost same amount of fiber and protein content as DDGS, with little deviations. The total starch content of DWG (overall average value of 11.01%) was quite similar to DDGS as well.

Conclusion

The main objective of this study was to examine various physical and chemical properties of DDGS, DDG, and DWG, along with image analysis and NMR spectroscopy. The main idea was to provide an overall picture of these coproducts, and how they could be used with value added alternatives such as enzymatic hydrolysis to yield additional ethanol, as biocomposites, as a livestock feed, or even for harvesting important precursor molecules. Least Significant Difference testing (LSD) was able to clearly show that there were differences in the properties between the three plants, as well as within the same plant across batches (i.e., time periods). Comparative view of the results of each property among DDGS, DDG, and DWG showed that DDGS would be a good choice over the other two materials for a variety of reasons. Thus, this research should provide a basis for pursuing other ways that DDGS could be used for value added products in future, and also what properties will be important. Key aspects of this study highlighted the necessity of optimization between various physical and chemical properties, and the need for consistency between batches.

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Table 1. Plant wise comparison of DDGS by LSD test (using α = 0.05).

Properties	Plant I	Plant II	Plant III
Moisture (% db)	4.61^{a}	4.98^{a}	5.18^{a}
Water activity	0.47^{b}	0.45^{b}	0.52^{a}
Bulk density (kg/m^3)	487.02^{b}	480.40^{b}	499.51^{a}
Thermal conductivity (W/m°C)	0.07^{a}	0.12^{a}	0.06^{a}
Thermal resistivity (m°C/W)	14.79^{ab}	14.69^{b}	15.50^{a}
Thermal diffusivity (mm^2/s)	0.14^{a}	0.14^{a}	0.15^{a}
Color (L)	40.29^{b}	43.42^{a}	43.68^{a}
Color (a)	9.37^{b}	18.71^{a}	12.15^{ab}
Color (b)	19.70^{a}	18.23^{a}	17.43^{a}
Angle of repose (°)	38.78^{a}	36.55^{a}	24.55^{b}
Ash (% db)	13.27^{a}	12.84^{a}	11.52^{a}
NDF (% db)	31.84^{b}	39.90^{a}	38.46^{a}
ADF (% db)	15.56^{a}	15.21^{a}	17.89^{a}
Crude fiber (% db)	9.93^{a}	10.30^{a}	10.32^{a}
Crude protein (% db)	28.33^{b}	30.65^{a}	29.70^{ab}
Crude fat (% db)	10.76^{a}	9.75^{a}	10.98^{a}
Total starch (% db)	11.82^{a}	9.81^{a}	11.59^{a}

Same letters for the plants indicates that they are not significantly different from each other for that property.

Table 2. Batch wise comparison of DDGS within plant I by LSD test (using α= 0.05).

Properties	Batch I	Batch 2
Moisture (% db)	4.61^{a}	4.91^{a}
Water activity	0.50^{a}	0.44^{b}
Bulk density (kg/m^3)	469.70^{b}	504.34^{a}
Thermal conductivity (W/m°C)	0.07^{a}	0.06^{a}
Thermal resistivity (m°C/W)	14.88^{a}	14.70^{a}
Thermal diffusivity (mm^2/s)	0.14^{a}	0.14^{a}
Color (L)	43.28^{a}	37.29^{b}
Color (a)	10.06^{a}	8.67^{b}
Color (b)	21.39^{a}	18.02^{b}
Angle of repose (°)	32.43^{b}	45.14^{a}
Ash (% db)	12.54^{a}	13.40^{a}
NDF (% db)	33.74^{a}	29.95^{b}
ADF (% db)	17.04^{a}	14.08^{a}
Crude fiber (% db)	9.63^{a}	10.22^{a}
Crude protein (% db)	29.20^{a}	27.45^{b}
Crude fat (% db)	11.15^{a}	10.40^{a}
Total starch (% db)	14.00^{a}	9.63^{b}

Same letters for the batches indicates that they are not significantly different from each other for that property

Table3. Batch wise comparison of DDGS within plant II by LSD test (using α= 0.05).

Properties	Batch I	Batch II
Moisture (% db)	4.60[a]	5.35[a]
Water activity	0.43[b]	0.48[a]
Bulk density (kg/m^3)	472.04[b]	488.76[a]
Thermal conductivity (W/m°C)	0.17[a]	0.07[a]
Thermal resistivity (m°C/W)	15.34[a]	14.04[a]
Thermal diffusivity (mm^2/s)	0.14[a]	0.13[a]
Color (L)	44.04[a]	42.45[a]
Color (a)	10.51[a]	26.91[a]
Color (b)	21.95[a]	12.91[a]
Angle of repose (°)	28.99[a]	44.11[b]
Ash (% db)	11.74[a]	11.30[a]
NDF (% db)	40.02[a]	39.80[a]
ADF (% db)	18.25[a]	12.71[b]
Crude fiber (% db)	9.31[b]	11.29[a]
Crude protein (% db)	29.85[b]	31.45[a]
Crude fat (% db)	9.15[a]	10.35[a]
Total starch (% db)	10.39[a]	9.23[a]

Same letters for the batches indicates that they are not significantly different from each other for that property.

Table 4. Batch wise comparison of DDGS within plant III by LSD test (using α= 0.05).

Properties	Batch I	Batch II
Moisture (% db)	4.98 [a]	5.38[a]
Water activity	0.53[a]	0.51[b]
Bulk density (kg/m^3)	490.41[b]	508.61[a]
Thermal conductivity (W/m°C)	0.06[a]	0.06[a]
Thermal resistivity (m°C/W)	15.24[a]	15.76[a]
Thermal diffusivity (mm^2/s)	0.14[a]	0.15[a]
Color (L)	43.14[a]	44.22[a]
Color (a)	10.47[a]	13.83[a]
Color (b)	21.53[a]	14.93[b]
Angle of repose (°)	16.63[b]	32.48[a]
Ash (% db)	16.59[a]	9.07[b]
NDF (% db)	39.85[a]	37.07[a]
ADF (% db)	17.83[a]	17.95[a]
Crude fiber (% db)	10.48[a]	10.15[a]
Crude protein (% db)	30.40[a]	29.00[b]
Crude fat (% db)	11.10[a]	10.85[a]
Total starch (% db)	12.61[a]	10.58[b]

Same letters for the batches indicates that they are not significantly different from each other for that property

Table 5. Plant wise comparison of DWG by LSD test (using α= 0.05).

Properties	Plant I	Plant II	Plant III
Moisture (% db)	33.17^{b}	29.36^{c}	40.10^{a}
Water activity	0.97^{a}	0.95^{b}	0.98^{a}
Bulk density (kg/m^3)	939.20^{b}	951.93^{b}	1076.16^{a}
Thermal conductivity (W/m°C)	0.12^{a}	0.13^{a}	0.14^{a}
Thermal resistivity (m°C/W)	9.49^{a}	8.61^{a}	10.00^{a}
Thermal diffusivity (mm^2/s)	0.11^{a}	0.11^{a}	0.12^{a}
Color (L)	57.46^{a}	46.81^{c}	54.66^{b}
Color (a)	6.56^{b}	8.10^{a}	5.97^{b}
Color (b)	25.25^{a}	22.23^{b}	23.57^{ab}
Ash (% db)	13.92^{a}	13.45^{a}	12.56^{a}
NDF (% db)	29.93^{c}	37.17^{a}	34.61^{b}
ADF (% db)	13.59^{a}	14.16^{a}	14.61^{a}
Crude fiber (% db)	12.23^{a}	12.00^{a}	11.91^{a}
Crude protein (% db)	29.85^{a}	27.13^{b}	28.83^{a}
Crude fat (% db)	9.70^{c}	10.93^{a}	12.75^{b}
Total starch (% db)	11.98^{a}	10.91^{a}	10.86^{a}

Same letters for the plants indicates that they are not significantly different from each other for that property

Table 6. Batch wise comparison of DWG within plant I by LSD test (using α= 0.05).

Properties	Batch I	Batch II
Moisture (% db)	33.24^{a}	33.10^{a}
Water activity	0.96^{a}	0.98^{a}
Bulk density (kg/m^3)	1032.44^{a}	845.95^{b}
Thermal conductivity (W/m°C)	0.16^{a}	0.09^{b}
Thermal resistivity (m°C/W)	6.70^{b}	12.28^{a}
Thermal diffusivity (mm^2/s)	0.10^{b}	0.12^{b}
Color (L)	56.28^{b}	58.63^{a}
Color (a)	6.23^{a}	6.89^{a}
Color (b)	24.87^{b}	25.62^{b}
Ash (% db)	14.15^{a}	13.68^{a}
NDF (% db)	30.84^{a}	29.02^{a}
ADF (% db)	14.42^{a}	12.77^{a}
Crude fiber (% db)	12.40^{a}	12.06^{a}
Crude protein (% db)	30.55^{a}	29.15^{b}
Crude fat (% db)	10.75^{a}	8.65^{b}
Total starch (% db)	10.54^{b}	13.42^{a}

Same letters for the batches indicates that they are not significantly different from each other for that property

Table 7. Batch wise comparison of DWG within plant II by LSD test (using α= 0.05).

Properties	Batch I	Batch II
Moisture (% db)	29.10[a]	29.62[a]
Water activity	0.95[a]	0.96[a]
Bulk density (kg/m^3)	1107.46[a]	796.40[b]
Thermal conductivity (W/m°C)	0.15[a]	0.12[a]
Thermal resistivity (m°C/W)	7.03[a]	10.19[a]
Thermal diffusivity (mm^2/s)	0.10[a]	0.12[a]
Color (L)	47.83[a]	45.78[a]
Color (a)	8.06[a]	8.16[a]
Color (b)	22.94[a]	21.52[b]
Ash (% db)	13.42[a]	13.48[a]
NDF (% db)	38.24[a]	36.10[a]
ADF (% db)	16.66[a]	11.65[b]
Crude fiber (% db)	11.99[a]	12.02[a]
Crude protein (% db)	26.45[b]	27.80[a]
Crude fat (% db)	12.85[a]	12.65[a]
Total starch (% db)	11.38[a]	10.43[b]

Same letters for the batches indicates that they are not significantly different from each other for that property

Table 8. Batch wise comparison of DWG within plant III by LSD test (using α= 0.05).

Properties	Batch I	Batch II
Moisture (% db)	39.58[a]	40.63[a]
Water activity	0.98[a]	0.98[b]
Bulk density (kg/m^3)	1078.16[a]	1074.17[a]
Thermal conductivity (W/m°C)	0.17[a]	0.12[a]
Thermal resistivity (m°C/W)	5.84[b]	14.18[a]
Thermal diffusivity (mm^2/s)	0.09[a]	0.14[a]
Color (L)	53.97[a]	55.35[a]
Color (a)	6.07[a]	5.86[a]
Color (b)	24.07[a]	23.08[a]
Ash (% db)	10.28[a]	14.83[a]
NDF (% db)	35.29[a]	33.32[b]
ADF (% db)	15.48[a]	13.74[a]
Crude fiber (% db)	12.16[a]	11.65[a]
Crude protein (% db)	28.00[b]	29.65[a]
Crude fat (% db)	10.85[a]	11.00[a]
Total starch (% db)	12.77[a]	8.95[b]

Same letters for the batches indicates that they are not significantly different from each other for that property

Table 9. Physical properties of DDGS (n=30 for each case*).*

Physical properties	Minimum	Maximum	Mean	Standard Deviation
Moisture Content (% db)	3.54	8.21	5.07	1.21
Water activity	0.42	0.53	0.48	0.04
Bulk density (kg/m^3)	467.7	509.38	488.97	14.96
Thermal Conductivity (W/m°C)	0.05	0.07	0.06	0.005
Thermal Resistivity (m°C/W)	13.1	16.4	14.88	0.037
Thermal diffusivity (mm^2/s)	0.1	0.17	0.17	0.008
Color (L)	36.56	50.17	42.3	2.99
Color (a)	5.2	10.79	9.65	1.23
Color (b)	12.53	23.36	20.62	2.93
Angle of repose (°)	25.7	47.04	45.14	11.37

Table 10. Physical properties of DDG (n=30 for each case*).*

Physical properties	Minimum	Maximum	Mean	Standard Deviation
Moisture (% db)	2.17	2.71	2.57	0.26
Water activity	0.42	0.42	0.42	0.00
Bulk density (kg/m^3)	467.3	482.24	472.03	5.85
Thermal Conductivity (W/m°C)	0.06	0.08	0.07	0.008
Thermal Resistivity (m°C/W)	12.8	17.9	15.72	2.24
Thermal diffusivity (mm^2/s)	0.14	0.17	0.15	0.02
Color (L)	52.43	54.82	53.97	0.90
Color (a)	5.9	6.72	6.07	0.13
Color (b)	23.76	24.29	24.07	0.20
Angle of repose (°)	20.32	29.9	23.88	3.73

Table 11. Physical properties of DWG (n=30 for each case).

Physical properties	Minimum	Maximum	Mean	Standard Deviation
Moisture (% db)	22.85	43.66	34.35	5.67
Water activity	0.93	0.99	0.96	0.02
Bulk Density (kg/m^3)	794.555	1107.6	989.10	123.78
Thermal Conductivity (W/m°C)	0.05	0.21	0.12	0.05
Thermal Resistivity (m°C/W)	4.74	16.4	9.66	4.17
Thermal diffusivity (mm^2/s)	0.08	0.24	0.11	0.03
Color (L)	42.04	57.31	50.94	4.167
Color (a)	3.41	8.88	6.91	1.185
Color (b)	14.89	27.73	23.75	2.32

Table12. Chemical properties of DDGS (n=30 for each case).

Chemical properties	Minimum	Maximum	Mean	Standard Deviation
Crude ash (% db)	5.00	21.93	12.82	3.19
NDF (% db)	26.32	43.50	36.74	4.46
ADF (% db)	10.82	20.05	16.2	4.22
Crude fiber (% db)	8.14	12.82	10.22	1.63
Crude protein (% db)**	27.4	31.7	29.93	1.30
Crude fat (% db)**	7.4	11.6	10.5	1.08
Total starch (% db)**	9.19	14.04	11.07	1.77

** no. of replications=2 for those properties.

Table 13. Chemical properties of DDG (n=30 for each case).

Chemical properties	Minimum	Maximum	Mean	Standard Deviation
Crude ash (% db)	8.84	11.43	10.91	1.53
NDF (% db)	29.39	33.82	31.43	0.64
ADF (% db)	27.99	29.42	28.69	1.63
Crude fiber (% db)	10.49	14.95	12.33	2.08
Crude protein (% db)**	30.6	31.2	30.9	0.42
Crude fat (% db)**	8.1	9.7	8.9	1.13
Total starch (% db)**	9.19	12.83	11.01	2.57

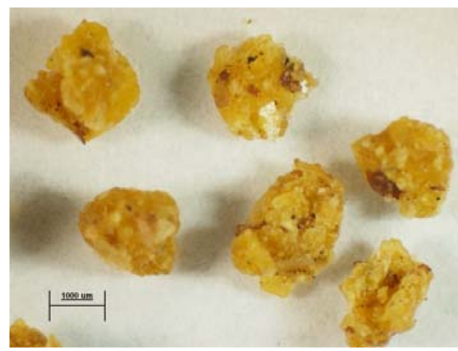
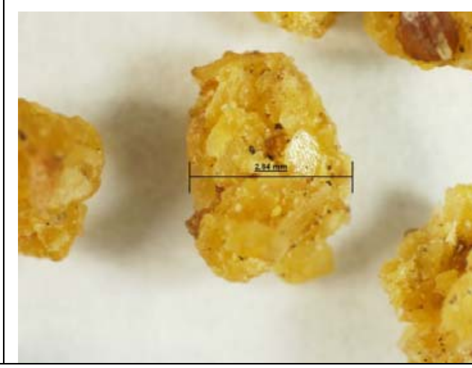
** no. of replications = 2 for those properties

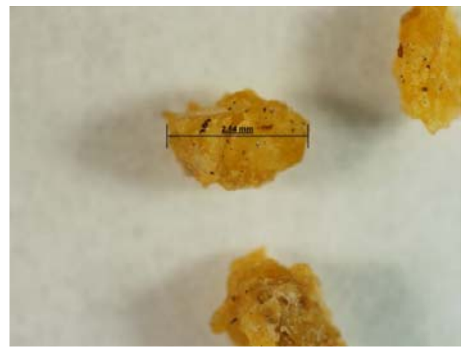
Table 14. Chemical properties of DWG (n=30 for each case).

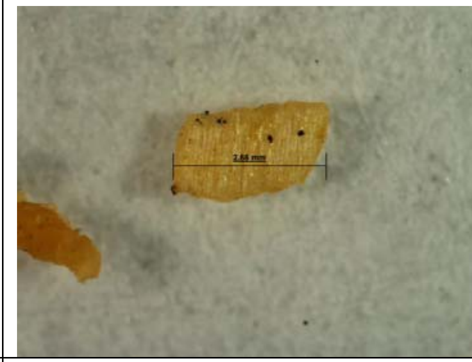
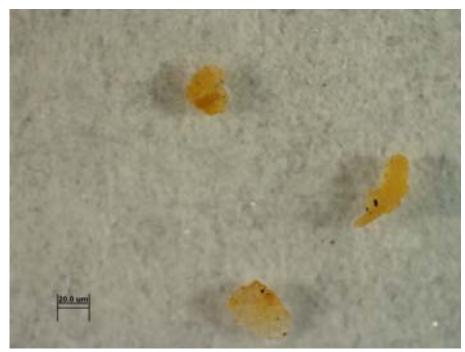
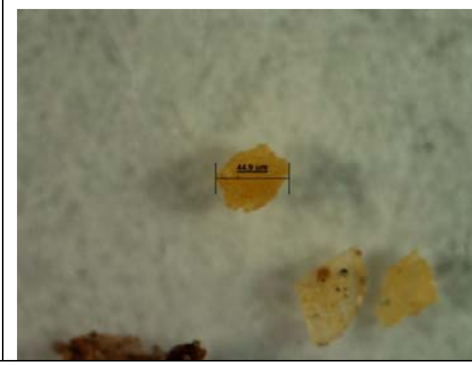
Chemical properties	Minimum	Maximum	Mean	Standard Deviation
Crude ash (% db)	8.11	22.29	13.31	3.15
NDF (% db)	27.07	40.06	33.80	3.63
ADF (% db)	12.85	15.04	14.22	2.04
Crude fiber (% db)	10.08	14.40	12.04	1.18
Crude protein (% db) **	26.3	30.6	28.62	1.44
Crude fat (% db) **	8.5	12.9	11.12	1.47
Total starch (% db) **	8.93	13.51	11.24	1.57

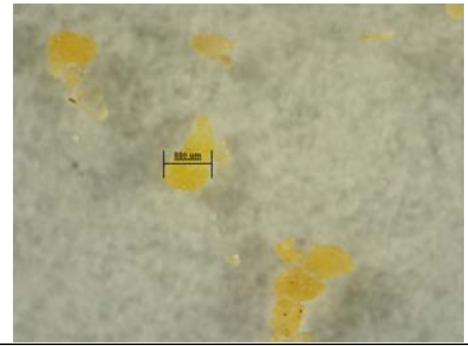
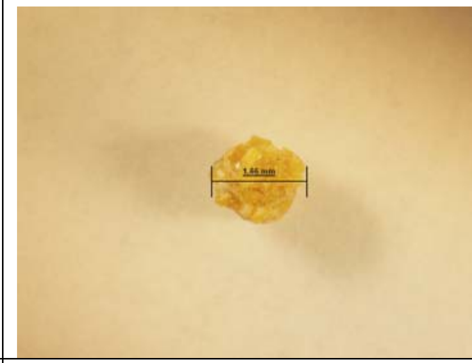
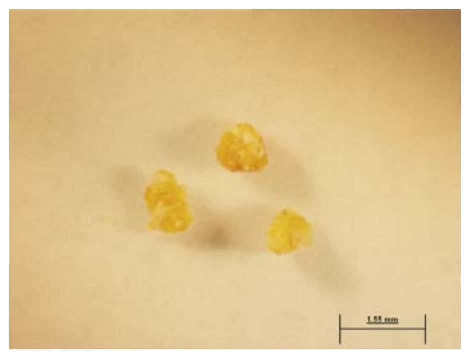
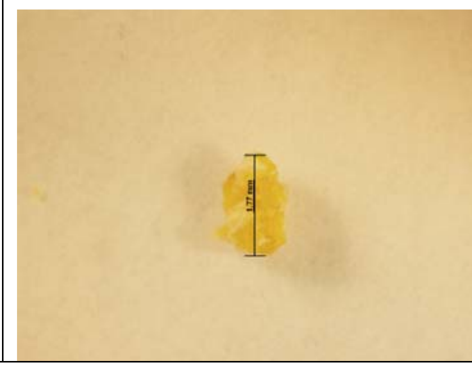
** no. of replications = 2 for those properties

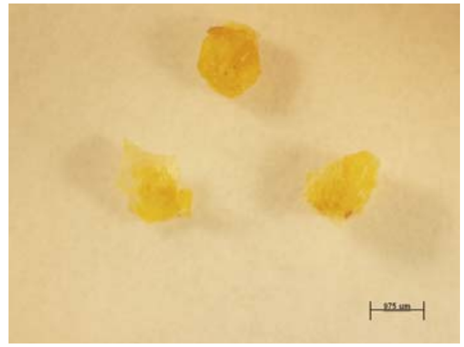
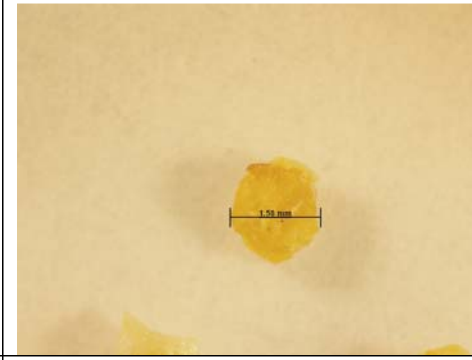
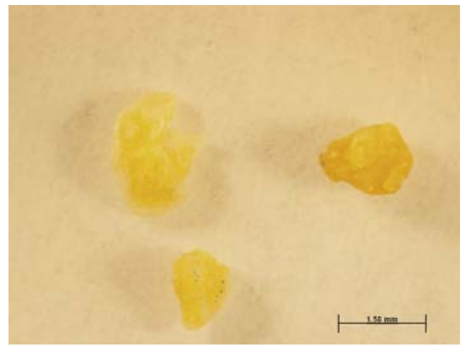
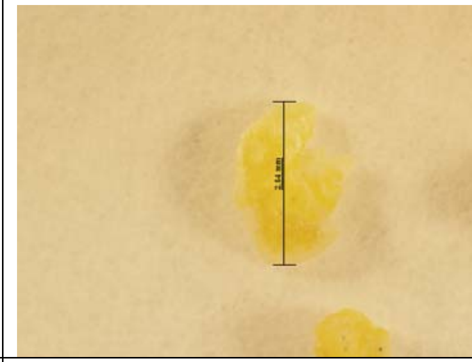
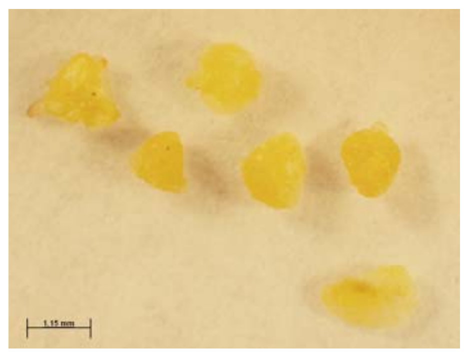
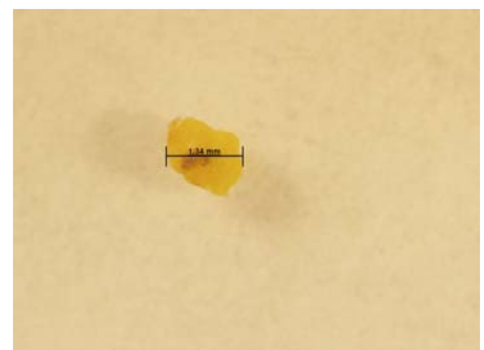
Table 15. Particle size and image analysis of DDGS.

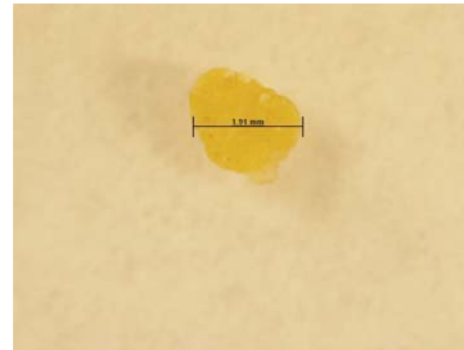
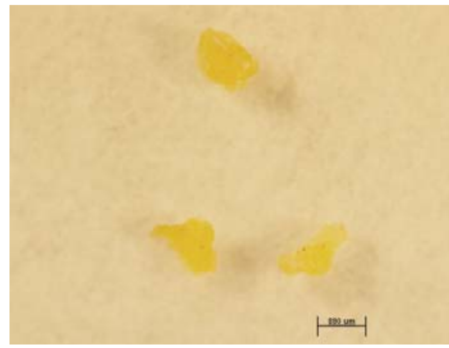
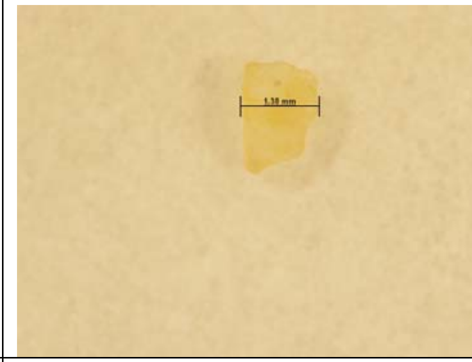
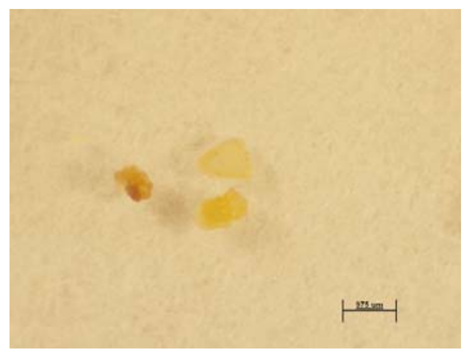
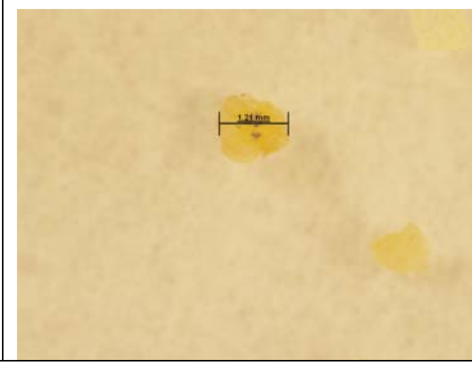
Plant	d_{gw} (mm)	S_{gw} (mm)	US sieve no.	Area (mm^2)	Max. diameter (mm)	Min. diameter (mm)	Round-ness (%)		
1	**0.85**	**0.51**		**5.11**	**6.87**	**0.17**	**64.30**		
			8	25.23	6.87	4.91	112.83		
			12	6.62	3.37	2.31	22.63		

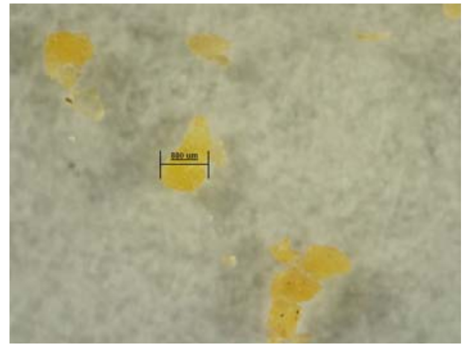
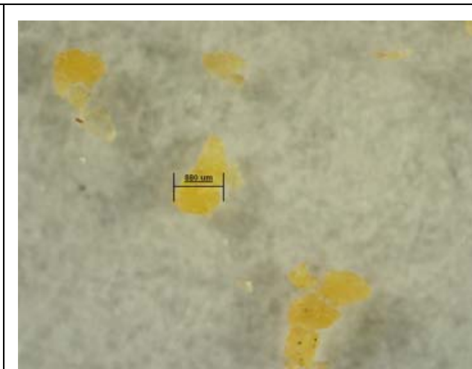
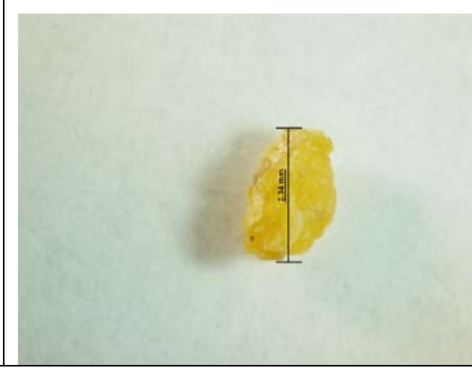
			16	6.30	1.29	0.79	63.46		
			20	2.46	0.95	0.72	64.86		
			30	2.03	0.62	0.44	224.66		

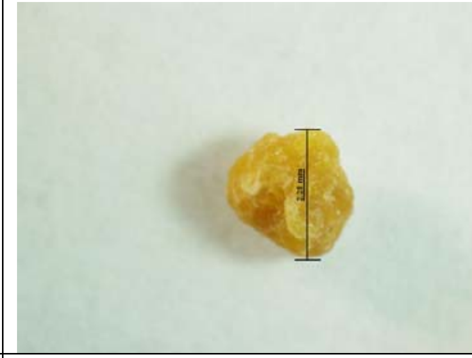
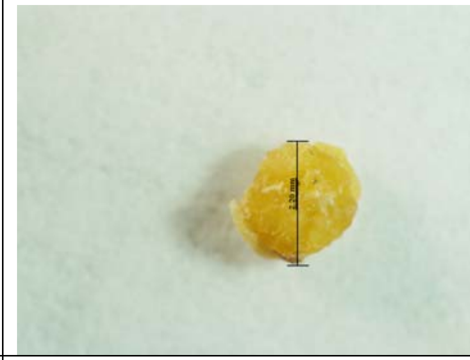
			40	1.22	0.41	0.40	56.71		
			50	0.95	0.45	0.24	47.18		
			70	0.71	0.41	0.26	34.16		

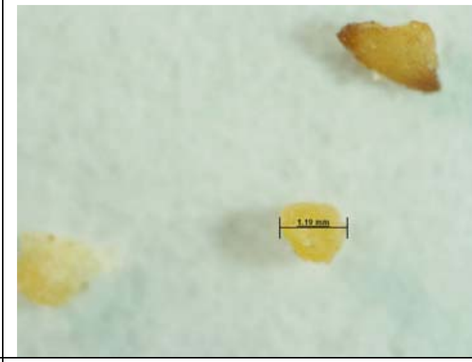
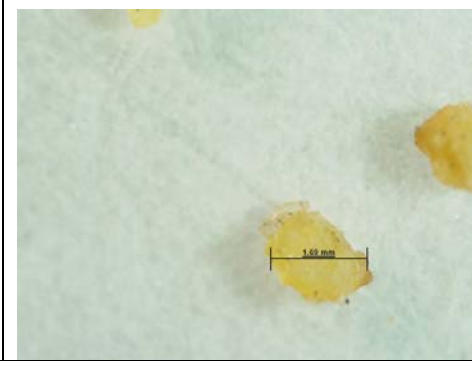
			100	0.52	0.37	0.17	17.09		
2	**0.69**	**0.37**		**5.66**	**6.40**	**0.50**	**31.30**		
			8	26.12	6.4	4.59	32.00		
			12	13.76	4.78	3.29	5.93		

			16	2.24	2.53	1.54	73.36		
			20	2.0	2.33	1.85	5.27		
			30	2.10	2.05	1.56	5.64		

			40	1.53	1.44	0.91	29.73		
			50	1.33	0.83	0.77	101.14		
			70	0.96	0.75	0.72	20.43		

			100	0.92	0.67	0.50	8.26		
3	**1.19**	**0.47**		**16.70**	**29.00**	**0.36**	**56.61**		
			8	43.57	29.00	19.62	248.00		
			12	33.4	17.80	12.49	108.48		

			16	27.14	7.15	5.37	15.46		
			20	11.75	2.88	1.84	27.63		
			30	10.85	1.97	1.04	40.92		

			40	9.59	0.93	0.89	25.95		
			50	8.18	0.86	0.74	14.36		
			70	4.5	0.70	0.58	10.11		

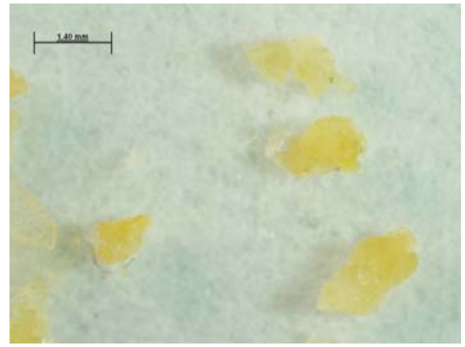
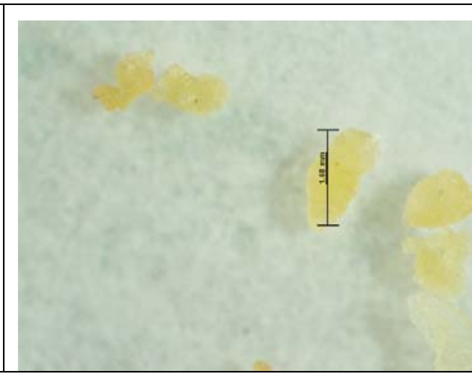
			100	1.06	0.45	0.36	18.65		

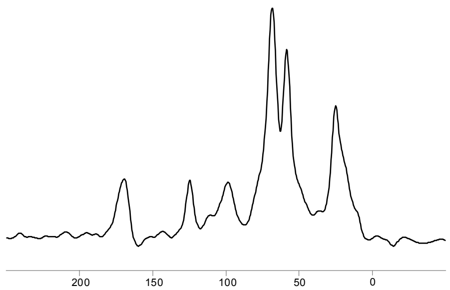
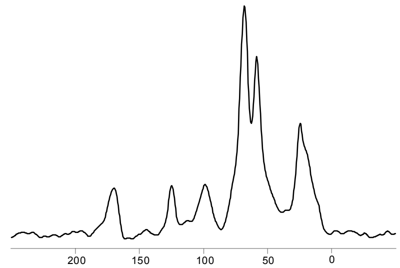
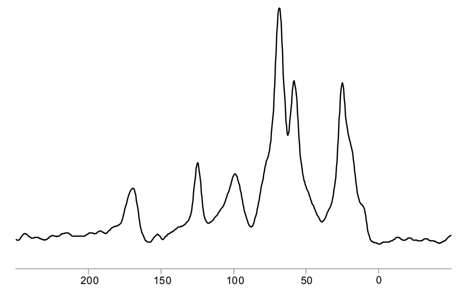
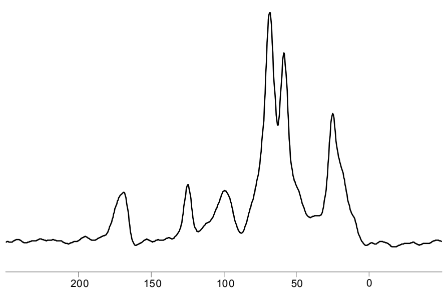
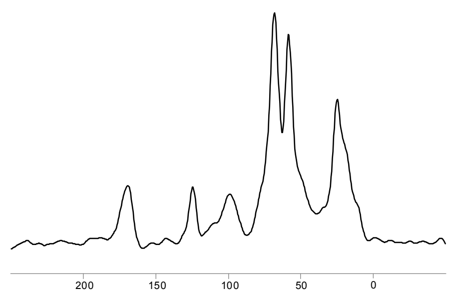
Table 16. Carbon partitioning (%) of DDGS.

Plant	Batch	Alkyl (0 – 50 ppm)	O-Alkyl (50 – 100 ppm)	Aromatic (100 – 160 ppm)	Carboxyl (160 – 190 ppm)	Carbonyl (190 – 220 ppm)
1	1	26.62	53.24	12.35	7.63	0.15
	2	27.92	54.59	10.41	6.99	0.07
2	1	26.81	54.33	11.55	7.19	0.09
	2	30.89	50.26	10.90	7.78	0.15
3	1	30.44	52.69	9.64	7.51	0.10
	2	30.46	51.57	9.66	8.24	0.05
Average		28.86	52.81	10.75	7.56	0.11
Standard deviation		1.96	1.66	1.07	0.44	0.04

Table 17. Carbon partitioning (%) of DDG.

Plant	Batch	Alkyl (0 – 50 ppm)	O-Alkyl (50 – 100 ppm)	Aromatic (100 – 160 ppm)	Carboxyl (160 – 190 ppm)	Carbonyl (190 – 220 ppm)
1	**1**	27.46	50.08	15.15	7.11	0.18

Table 18. NMR spectra results for DDGS and DDG.

	DDGS		DDG
Plant	**Batch I**	**Batch II**	**Batch I**
I			
II	**Batch I**	**Batch II**	
			
III	**Batch I**	**Batch II**